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National Aeronautics
and Space Administration

**Scientific and Technical
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1978

EFFECTIVE THERMAL CONDUCTIVITY DETERMINATION

FOR LOW-DENSITY INSULATING MATERIALS

By S. D. Williams* and Donald M. Curry
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SUMMARY

A nonlinear least squares technique has been used to determine effective thermal conductivity values using experimental data for a low-density insulating material. A comparison of the analytically predicted conductivity with values obtained from standard guarded-hot-plate tests indicates that the predicted values can be used with confidence in performing thermal analyses. An assessment of the predicted thermal conductivity, from a statistical viewpoint, and of its effect on the thermal response of the material was performed and is discussed herein. Results of this investigation demonstrate that thermal conductivity values can be routinely predicted using analytical methods rather than being obtained through expensive, time-consuming standard guarded-hot-plate tests.

INTRODUCTION

The design and development of a reusable thermal protection system (TPS) for the Space Shuttle Orbiter required a detailed investigation of various material classes and configurations, and a knowledge of the thermophysical properties of these materials. Since direct measurement of effective thermal conductivity is both expensive and subject to inaccuracy, guarded-hot-plate (GHP) data may be supplemented with effective conductivity inferred from in-depth thermocouple (TC) temperature histories obtained from thermal evaluation tests (plasma arc jet/radiant). Using analytical methods, properties can be routinely derived from these types of tests rather than being obtained from expensive, time-consuming standard tests.

Of special interest are analytical methods that permit the evaluation of thermal response data obtained from a single experiment. Numerical techniques have been developed to determine thermophysical properties and surface temperature response from in-depth temperature measurements. In particular, Curry and Williams (ref. 1) have shown that meaningful effective thermal conductivity can be obtained by use of nonlinear least squares techniques

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from thermal tests not specifically designed to measure the thermal conductivity. Banas and Cunnington (ref. 2) have discussed laboratory measurements of effective thermal conductivity, fiber diameter, and extinction coefficient for the all-silica, low-density, rigid insulation material selected for the Orbiter TPS.

The application of the nonlinear least squares method in calculating effective thermal conductivity values using transient thermal response data is discussed in this paper. A comparison of the analytically predicted conductivity with values obtained from a standardized GHP apparatus (ref. 3), is presented. An assessment of the predicted effective thermal conductivity, from a statistical viewpoint, and of its effect on the thermal response of the material is also discussed. In addition, the results obtained from using a Space Shuttle mission profile response are given. These simulations demonstrate that reliable results can be obtained using derived conductivity values rather than GHP data.

As an aid to the reader, where necessary the original units of measure have been converted to the equivalent value in the Système International d'Unités (SI). The SI units are written first, and the original units are written parenthetically thereafter.

SYMBOLS

a_0, a_1, a_2, a_3	coefficients in conductivity polynomial (eqs. (2) and (5))
dY_i	error between predicted and measured thermocouple data (eq. (8))
k_c	conductivity due to conduction and convection (eq. (1))
k_e	effective thermal conductivity (eq. (1))
k_R	conductivity due to radiation (eq. (1))
ℓ	degree of polynomial fit (eq. (8))
m	number of time measurements (eq. (3))
n	number of thermocouples (eq. (3))
N	number of baseline conductivity values (eq. (9))
p	number of measurements taken with a thermocouple (eq. (8))
Q_{in}	input heat (fig. 2)
r	level of confidence (eq. (7))
S	sum of squares (eq. (3))

T	temperature (eq. (2))
$T(t)$	temperature function (fig. 2)
T_{ij}	predicted temperature at i-th thermocouple and j-th time measurement (eq. (3))
T_{ij}^*	measured temperature at i-th thermocouple and j-th time measurement (eq. (3))
x	depth of test article (fig. 2)
Δk_q	difference between baseline conductivity and predicted effective conductivity (eq. (9))
$\overline{\Delta k}$	statistical mean of Δk_q (eq. (9))
σ_k	standard error of estimate of conductivity (eq. (5))
$\tilde{\sigma}_k$	standard deviation of conductivity between baseline and predicted effective conductivity (eq. (9))
σ_T	standard error of estimate of temperature (eq. (4))
$\tilde{\sigma}_T$	standard error of estimate of temperature for thermocouple data (eq. (8))
Subscripts	
i	thermocouple measurement
j	time measurement

THEORETICAL FORMULATION

To evaluate an effective method for determining thermal property values, it is necessary to establish the fitting equations that are to be used. The purposes of the curve-fitting equations are to summarize a large quantity of data, to establish interpolation equations, and to confirm the analytical model.

Since it may be difficult to obtain explicit comparisons with the fitting equations, criteria should be established to satisfy the analyst that the results are reliable. The following criteria were used to ensure a good fitting method.

1. Use all of the relevant data in estimating each constant.
2. Have reasonable economy in the number of constants required.

3. Provide some estimate of the uncontrolled error in the prediction.
4. Make it possible to find regions of systematic deviations or functional bias from the equation, if any exist.
5. Show whether the conclusions are unduly sensitive to the results of a small number of runs, perhaps even to one run.
6. Give some idea of how well the final equation can be expected to predict the response, both in the overall sense and for important sets of conditions inside the region covered by the data.

The material investigated in this study is a low-density (144.2 kg/m³), rigid, fibrous insulation with a high porosity (94 percent void volume) and with transparency that permits heat transfer to occur by conduction, convection, and radiation. The effective thermal conductivity k_e , discussed in this paper can be expressed as

$$k_e = k_c + k_R \quad (1)$$

where k_c is the conductivity due to conduction and convection, and k_R is the apparent conductivity due to internal radiation transfer. In this investigation, it is assumed that the effective thermal conductivity can be expressed as a polynomial in temperature

$$k_e = a_0 + a_1T + a_2T^2 + a_3T^3 \quad (2)$$

where the values of the unknown coefficient a_j are to be determined for a minimal difference between the measured temperatures and the predicted temperatures. If T_{ij} represents the predicted temperature at the i -th thermocouple and the j -th time measurement, the least squares problem is to minimize

$$S(a_0, a_1, a_2, a_3) = \sum_{j=1}^m \sum_{i=1}^n (T_{ij} - T_{ij}^*)^2 \quad (3)$$

where S is the sum of squares, m is the number of time measurements, n is the number of thermocouples, and T_{ij}^* is the measured temperature. Reference 1 provides the justification for selecting the model in this form and demonstrates that the most efficient technique in solving for the unknown

coefficients is one developed by Peckham (ref. 4). It is obvious that the first criterion is satisfied by the selection of thermocouple locations and the choice of times during the test at which data will be selected for fitting. Since only four coefficients are used, the second criterion is also satisfied.

The third criterion can be satisfied by examining the converged least squares fit. A statistical measurement can be made to determine the accuracy of the converged effective conductivity polynomial. This analysis is given in the form of the standard error of estimates of temperature σ_T and conductivity σ_k from the least squares fit of the data. The standard error of temperature is given by

$$\sigma_t = \left(\frac{S}{mn - 4} \right)^{\frac{1}{2}} \quad (4)$$

where the number 4 accounts for the number of coefficients used to fit the data. To determine the standard error of conductivity from σ_T and the representation for conductivity, one has

$$\sigma_k = \sigma_t \left| \frac{\partial k_e}{\partial T} \right| = \sigma_t \left| a_1 + 2a_2T + 3a_3T^2 \right| \quad (5)$$

It has been found that the values of σ_k alone offer little meaning to the analyst and cannot be effectively used to satisfy the fourth criterion. However, if one uses σ_k as a measure of the accuracy of k_e , the information is useful. A convenient method is to use σ_k as a percent error of the effective conductivity; thus,

$$\text{Percent error} = \frac{\sigma_k}{k_e} \times 100 \quad (6)$$

This method can also be used to establish confidence limits on the accuracy of the data fit. Since there is no guarantee of the least squares fit being normally distributed, one must use the more conservative Chebyshev inequality (ref. 5)

$$\pm r\sigma_k \geq \left(1 - \frac{1}{r^2}\right) \times 100 \quad (7)$$

where r is the level of confidence. In terms of the percent error expressed in equation (6), this expression means that a 3σ confidence level (90 percent) on the accuracy can be related in terms of conductivity. Thus, if one has a 3-percent error in conductivity, he can be reasonably assured that a 9-percent error will bound 90 percent of the cases investigated.

Test Data Reduction

In the least squares procedure, it is assumed that the independent variables are measured without error. However, the data resulting from thermal simulation tests always contain a certain amount of noise and usually have a few "wild" points (i.e., data values that are so far removed from the trend of the other data that they are obviously invalid). To the analyst, these irregularities present no problem since the data are simply plotted at intervals much greater than the sample rate, and obvious wild points are discarded. Synthesis of the thermal response data requires that the computer program accept raw thermocouple data as input, usually from a magnetic tape. Difficulties arise, however, when the raw thermocouple data are used since, during the course of the repetitive data reduction and thermal calculations, even small errors can be magnified to make the output meaningless.

An algorithm has been developed to smooth the thermocouple data by fitting various least squares polynomials on overlapping data subsets. An attempt to fit all of the temperature data with a single polynomial would lead to large errors since a single polynomial cannot be found that represents all of the data. In addition, the fit at the end points would be extremely poor.

Exhaustive numerical testing has shown that the best results can be obtained by using a seventh-degree polynomial on each segment for the least squares fit. When all data are collected, the standard error of temperature $\tilde{\sigma}_T$ can be computed from

$$\tilde{\sigma}_T = \left[\sum_{i=1}^p \frac{(dY_i)^2}{p - \ell} \right]^{\frac{1}{2}} \quad (8)$$

where dY_i is the error computed, p is the number of thermocouple data measurements, and ℓ is the degree of the polynomial fit. The standard

error of temperature $\tilde{\sigma}_T$ can be used to accept or reject thermocouple data and thereby to ensure use of only those data that will produce meaningful results.

Thermocouple Location Errors

It is often difficult to physically locate accurately the position of the thermocouples; however, in predicting thermal conductivity by means of the mathematical model, these locations are assumed to be exact. After converging to the least squares approximation, the nonlinear least squares (NONLIN) program attempts to relocate the thermocouples in order to reduce the least squares errors. Frequently, this procedure alters the conductivity values such that they become more representative of the actual conductivity. In addition, by analyzing the results from the relocated thermocouples, the fifth criterion can be satisfied in an economical manner.

Prediction Technique Validation

The validation of the predicted effective thermal conductivity values can be established by several procedures. The most obvious method is to use the results to predict in-depth thermal responses and compare them with measured data. The next procedure is to use the conductivity values obtained from different constant-pressure tests to predict the in-depth response of the material subjected to variable heating and pressure conditions. The final validation consists of using the conductivity values obtained from the test specimen to predict the in-depth response of additional test specimens. These three validating procedures will satisfy the sixth criterion.

EXPERIMENTAL/ANALYTICAL VERIFICATION

A series of thermal tests using the Space Shuttle Orbiter reusable surface insulation (RSI) material was performed to validate the design thermal properties.

Test Conditions

The tests consisted of exposing the RSI TPS test article to steady-state temperature conditions of 1367 K at various constant pressures over a range from 0.10 to 101 kN/m² (0.76 to 760 torr). In addition, the test articles were exposed to transient trajectory temperature and flight pressure profile conditions. The test chamber was evacuated to the desired test pressure condition for a minimum of 1 hour before initiation of the heating cycle. Each test cycle was initiated when all thermocouples were essentially at the same temperature. Data were taken for a minimum of 1 hour after initiation of the heating cycle. Heater operating conditions, environmental test conditions, and model temperature response data were recorded on magnetic

tape on an analog-to-digital recording system. The data on the tapes were reduced to engineering units by use of a Univac 1100 series computer system.

Test Article Configuration

A cross-sectional view of the test article/test fixture is shown in figure 1. Two RSI tiles (15.24 by 15.24 by 11.684 cm), which were fabricated using baseline silica fibers and were coated on the top surfaces and side-walls with a reaction-cured glass (RCG) formulation, were provided by the Lockheed Missiles and Space Company. Each tile contained a 3.81-cm-diameter plug that was instrumented with six 127-micrometer (5 mil) platinum/platinum/13-percent-rhodium thermocouples in the center of the tile.

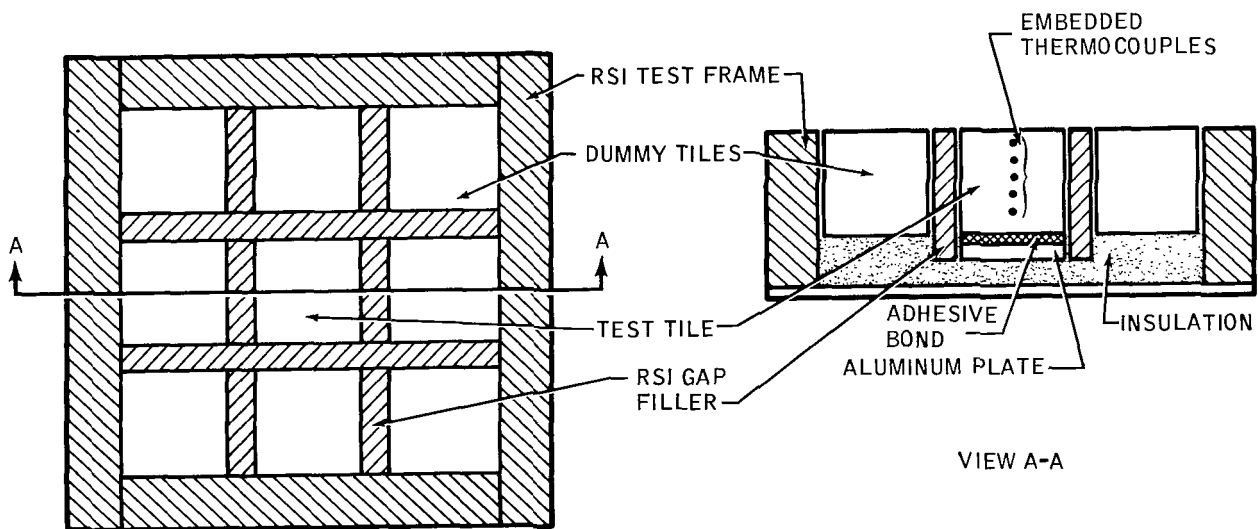


Figure 1.- Cross-sectional view of the RSI test article/test fixture.

Effective Thermal Conductivity Predictions

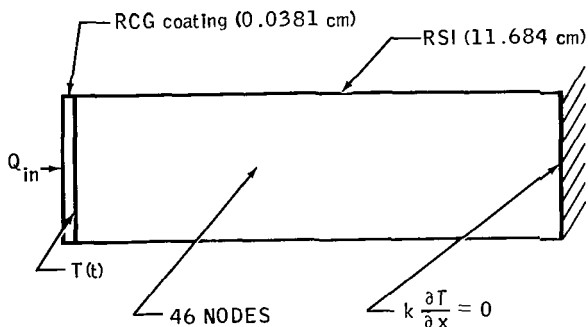


Figure 2.- Thermal model used to simulate test article configuration.

The NONLIN program was used to calculate effective thermal conductivity values using the thermal response data acquired from the constant-pressure/radiant-heating tests. The results of each radiant test were analyzed using a one-dimensional thermal mathematical model (fig. 2) developed to simulate the test article configuration. Since it is difficult to locate the exact thermocouple position, the NONLIN program, after converging to the least squares approximation, assesses positional error

effects and then repositions the thermocouples analytically to reduce the σ_T value.

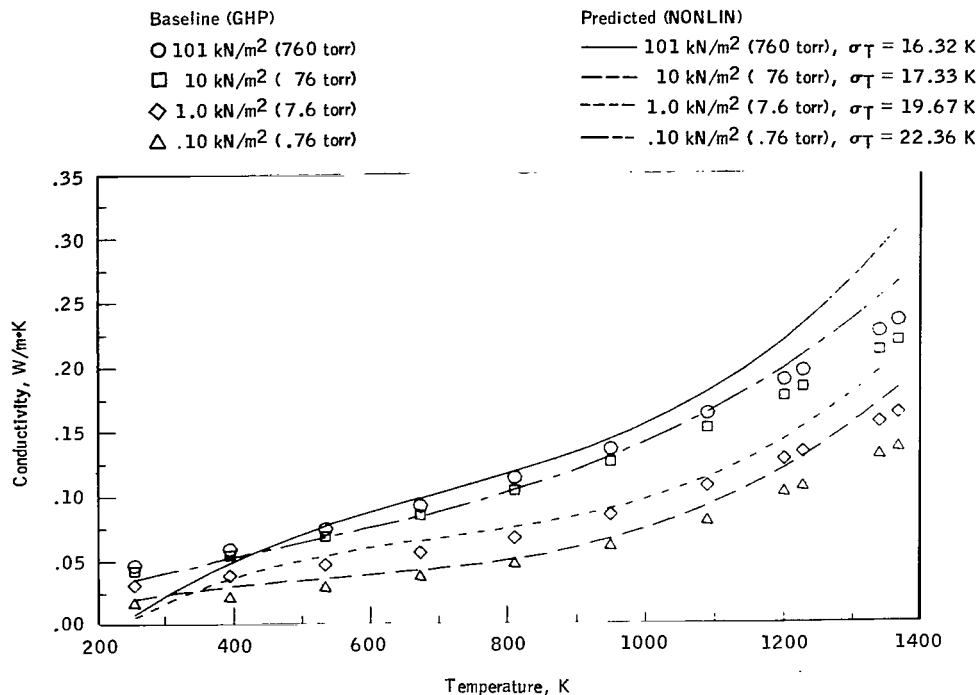
A composite plot of the predicted effective thermal conductivity as a function of temperature (with pressures of 101, 10, 1.0, and 0.10 kN/m² (760, 76, 7.6, and 0.76 torr)) using the steady-state test data with nominal and relocated-thermocouple locations for specimen RB37 can be seen in figures 3(a) and 3(b), respectively. A comparison with baseline thermal conductivity values is also indicated in these figures. These baseline values were obtained using a GHP apparatus with earlier material samples. The effect of using the relocated-thermocouple locations, in this case, was to reduce the values of σ_T by 50 percent (table I) and to raise the conductivity values slightly. A comparison of the calculated thermocouple depths with the nominal values can be seen in table II. By relocating the thermocouples, the percent error based on σ_k also was reduced by more than 50 percent from a typical value of 5 percent to 2 percent. (See figs. 4(a) and 4(b).) This result indicates that a 3σ confidence level of less than 6 percent error can be anticipated by using relocated data values. It is interesting to observe that the percent error for 101-kN/m² (760 torr) and 1.0-kN/m² (7.6 torr) data is sufficiently large in the same region (less than 450 K) that an analyst would interpret the conductivity data as being subject to error. In addition, the values for σ_T are sufficiently large to make the data suspect. In general, the predicted conductivity values are approximately 18 percent higher than the GHP data¹. However, the predicted conductivity values for pressures of 101 and 1.0 kN/m² (760 and 7.6 torr) and temperatures less than 450 K are less than the GHP values. This reversal in calculated conductivity is not consistent with the other results but is explained by the larger deviation between measured and predicted temperature for thermocouples 5 and 6 at 101 and 1.0 kN/m² (760 and 7.6 torr). The results for thermocouples 5 and 6 are also consistent with the error deviations shown in table III between the raw test data and the least squares polynomials. Table IV shows the standard deviation of conductivity $\tilde{\sigma}_k$ between the baseline (GHP) and predicted effective thermal conductivity using both nominal and relocated-thermocouple depths.

The standard deviation $\tilde{\sigma}_k$ is defined as

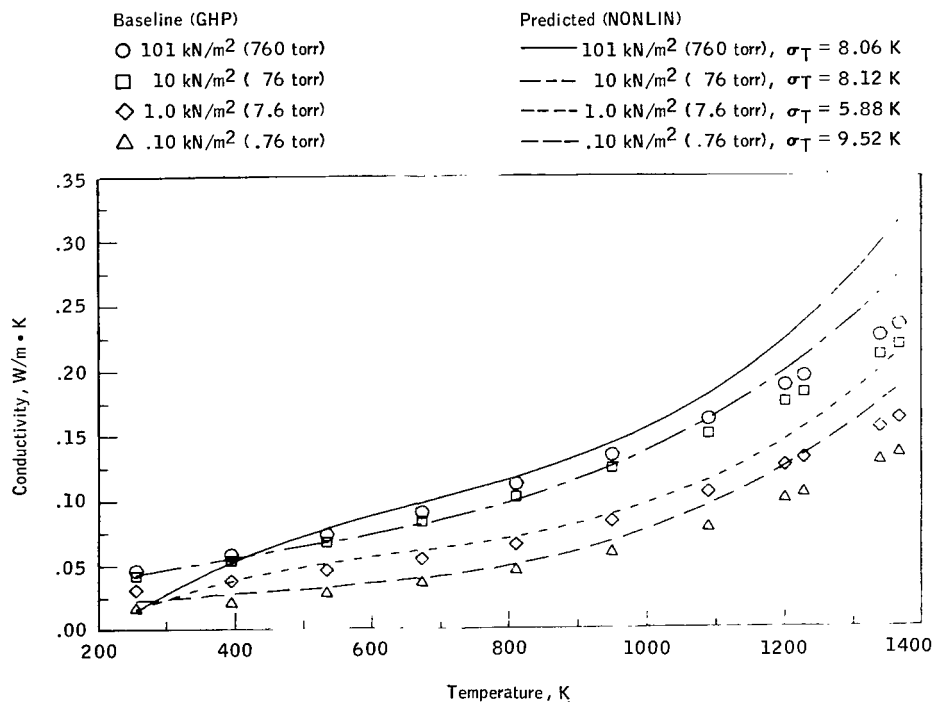
$$\tilde{\sigma}_k = \left[\frac{\sum_{q=1}^N (\Delta k_q - \overline{\Delta k})^2}{N - 1} \right]^{\frac{1}{2}} \quad (9)$$

Figure 3.- Comparison of predicted thermal conductivity for specimen RB37 (curves) with baseline conductivity values (data points). (overleaf)

¹The GHP tolerances on the RSI thermal conductivity are ± 18 percent.



(a) Nominal thermocouple locations.



(b) Relocated-thermocouple locations.

TABLE I.- A COMPARISON OF THE STANDARD ERROR OF ESTIMATE
OF TEMPERATURE FOR NOMINAL AND RELOCATED-THERMOCOUPLE LOCATIONS

Pressure, kN/m ² (torr)	σ_T , K	
	Nominal TC	Relocated TC
101.32 (760.00)	16.32	8.06
10.13 (76.00)	17.33	8.12
1.01 (7.60)	19.67	5.88
.10 (.76)	22.36	9.52

TABLE II.- A COMPARISON OF NOMINAL AND RELOCATED-THERMOCOUPLE DEPTHS

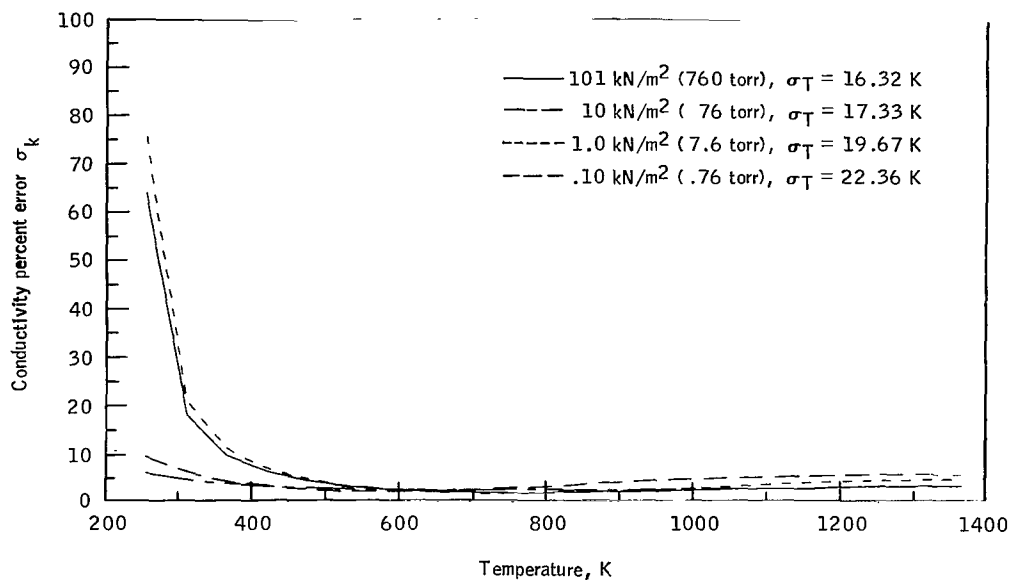
Specimen	Pressure, kN/m ² (torr)	Type (a)	Thermocouple depths, cm					
			TC ₁	TC ₂	TC ₃	TC ₄	TC ₅	TC ₆
RB37	101.32 (760.00)	R	0.0381	0.376403	0.997356	1.636344	2.747848	5.456098
RB37	10.13 (76.00)	R	.0381	.378917	1.002817	1.630782	2.699004	5.473497
RB37	1.01 (7.60)	R	.0381	.389966	1.013130	1.611935	2.710358	5.510886
RB37	.10 (.76)	R	.0381	.391744	1.016813	1.634566	2.668194	5.223713
RB37	--	N	.0381	b.4572	c.9398	b1.6256	d2.7686	d5.3086
B5892	--	N	.0381	b.4572	d1.5494	d3.8608	d6.5786	c9.0424

^aR = relocated-thermocouple values; N = nominal values.

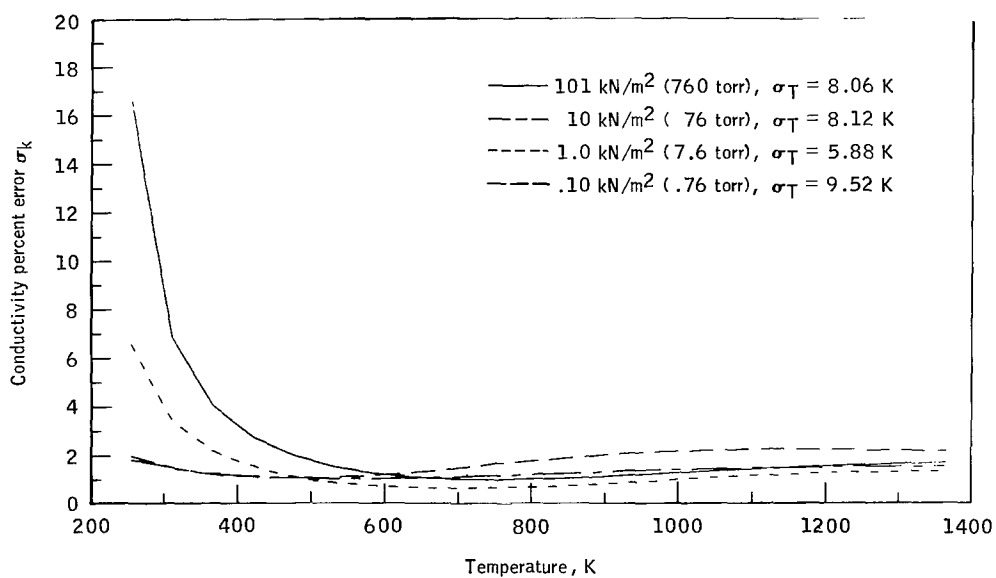
^bPlus or minus 0.03 centimeter.

^cPlus or minus 0.09 centimeter.

^dPlus or minus 0.05 centimeter.



(a) Nominal thermocouple locations.



(b) Relocated-thermocouple locations.

Figure 4.- Conductivity percent error based on σ_k .

TABLE III.- A STANDARD ERROR OF TEMPERATURE FOR LEAST SQUARES FITTING
OF THERMOCOUPLE DATA FOR SPECIMEN RB37

Pressure, kN/m ² (torr)	Standard error of temperature, K					
	TC ₁	TC ₂	TC ₃	TC ₄	TC ₅	TC ₆
101.32 (760.00)	0.646	3.335	1.950	2.422	11.213	5.878
10.13 (76.00)	.343	2.749	1.764	2.034	9.966	5.544
1.01 (7.60)	.542	3.763	3.151	3.151	13.202	7.197
.10 (.76)	.511	3.231	2.058	2.448	12.723	6.182

TABLE IV.- STANDARD DEVIATION OF THERMAL CONDUCTIVITY

Pressure, kN/m ² (torr)	$\tilde{\sigma}_k$, W/m·K	
	Nominal TC	Relocated TC
101.32 (760.00)	0.0315	0.0330
10.13 (76.00)	.0182	.0206
1.01 (7.60)	.0192	.0190
.10 (.76)	.0150	.0169

where N is the number of baseline conductivity values, Δk_g is the difference between GHP and predicted conductivity values, and $\overline{\Delta k}$ is the statistical mean of Δk_g . Although the $\tilde{\sigma}_k$ values obtained by using the nominal data are slightly less than those obtained by means of the relocated

data, the temperature errors are greater. The differences between the $\bar{\sigma}_k$ values for the nominal and relocated data are not significant from a statistical viewpoint; however, the differences in σ_T values are significant.

Figures 5(a) to 5(d) present a comparison of measured and predicted in-depth temperatures for constant pressures of 0.10, 1.0, 10, and 101 kN/m² (0.76, 7.6, 76 and 760 torr), respectively, using nominal thermocouple locations and the corresponding predicted conductivity values for specimen RB37. Figures 6(a) to 6(d) present the same comparison of temperatures using relocated-thermocouple locations and the corresponding predicted conductivity values for specimen RB37. The predictions using the relocated data are in closer agreement with the measured data, which confirms the analysis based on both σ_T and σ_k .

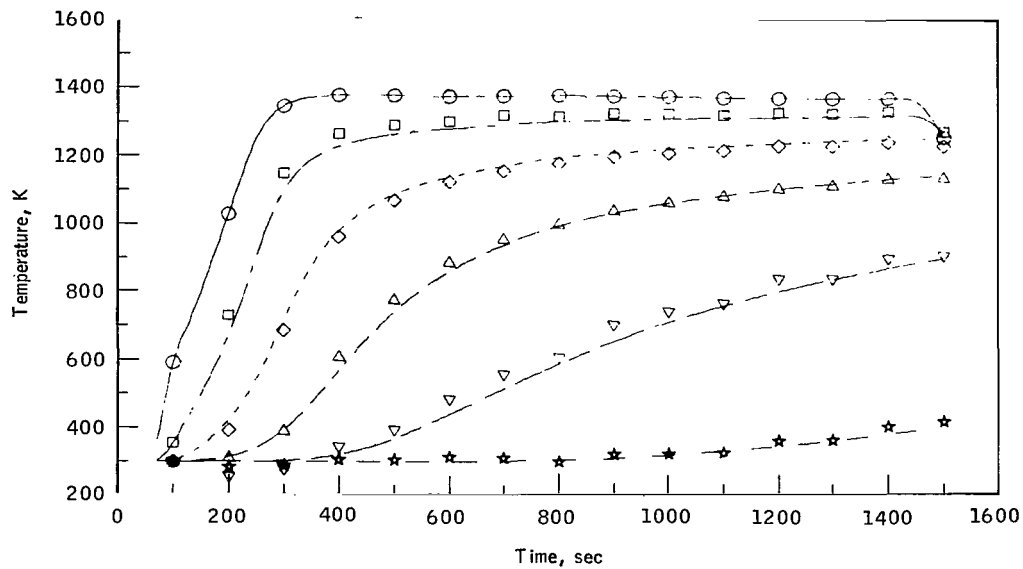
Because of the errors indicated in the predicted conductivity values (figs. 4(a) and 4(b)), engineering judgment was used to "fair" the conductivity values at pressures of 101 and 1.0 kN/m² (760 and 7.6 torr). The same thermal response predictions were made as previously discussed, and the results agreed well with those presented in figures 5 and 6. This result confirmed that the data in this region were not significant in the thermal conductivity estimation.

Thermal response predictions for the mission profile (variable surface temperature and pressure) using the predicted effective thermal conductivity and the baseline conductivity (GHP) for specimen RB37 are shown in figures 7(a) and 7(b), respectively. As can be seen, a better comparison between measured and predicted in-depth temperatures is obtained using the analytically determined conductivity. The predicted thermal response for the last two thermocouples (5 and 6) was lower than anticipated on the basis of the results of the constant-temperature/constant-pressure tests. This result is attributed to the noise present for the first 600 seconds on all tests for these two thermocouples.

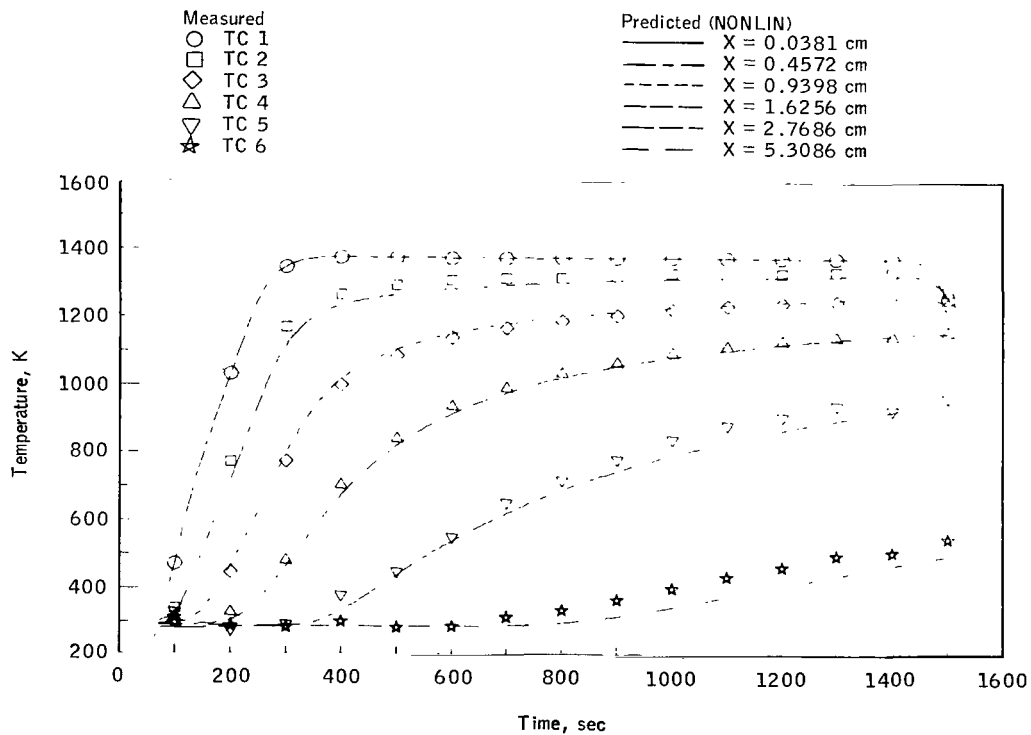
Thermal response predictions for the mission profile using the predicted effective thermal conductivity and the baseline conductivity (GHP) for the second test article (specimen B5892) are shown in figures 8(a) and 8(b), respectively. The nominal thermocouple depths for specimen B5892 are given in table II. As before, a better comparison between measured and predicted in-depth temperatures is obtained using the analytically determined conductivity.

These mission simulation tests demonstrate the uniqueness of the inferred effective conductivity values obtained from constant-surface-temperature/constant-pressure tests. In addition, these comparisons demonstrate that more reliable results for low-density, high-porosity, and semitransparent insulative materials can be obtained by using analytically calculated effective conductivity values than can be obtained by using GHP data. The reasons for these better predictions using effective conductivity appears to be due to several factors.

1. The GHP values are obtained from steady-state conditions and use a mean temperature in calculations for conductivity, whereas the analytical methods depend on local gradients in a transient environment.

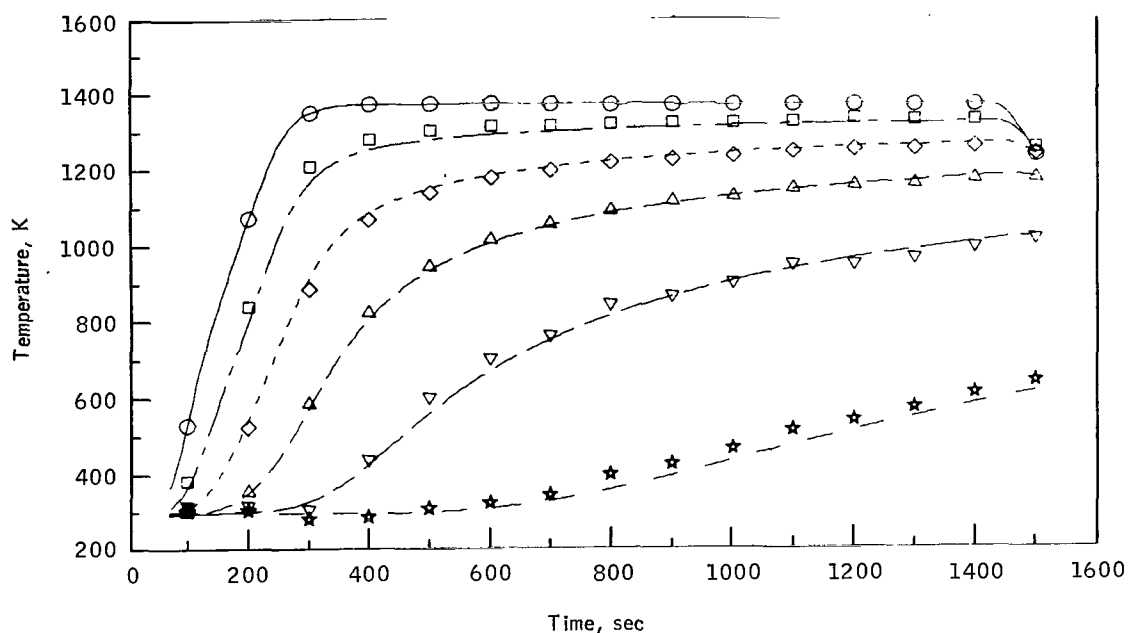


(a) 0.10 kN/m^2 (0.76 torr).



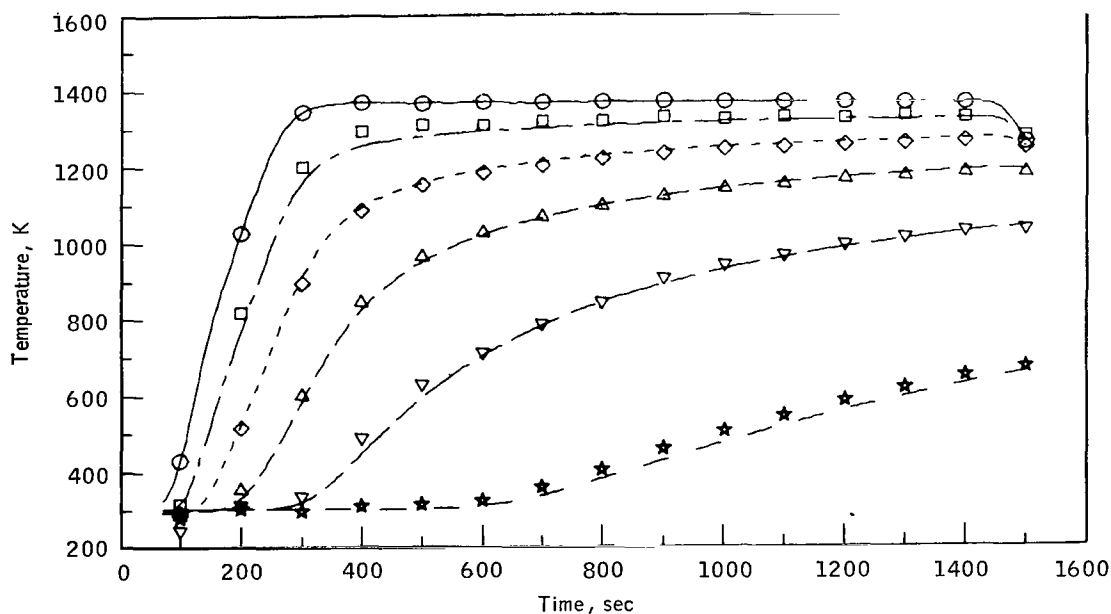
(b) 1.0 kN/m^2 (7.6 torr).

Figure 5.- Comparison of temperature response using predicted thermal conductivity from specimen RB37 tested at various pressures for nominal thermocouple (TC) locations at depth X (curves) with measured thermocouple values (data points).



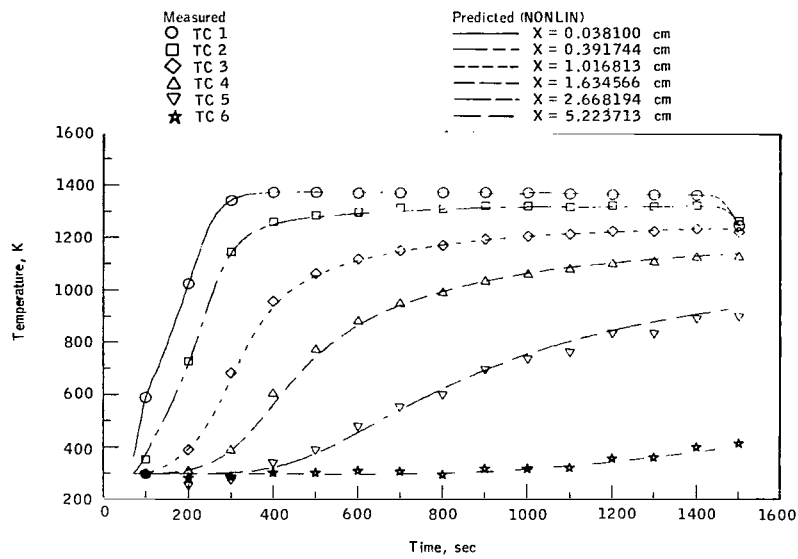
Measured
 ○ TC 1
 □ TC 2
 ◇ TC 3
 △ TC 4
 ▽ TC 5
 ★ TC 6

Predicted (NONLIN)
 — X = 0.0381 cm
 - - X = 0.4572 cm
 - - - X = 0.9398 cm
 - - - X = 1.6256 cm
 - - - X = 2.7686 cm
 - - - X = 5.3086 cm

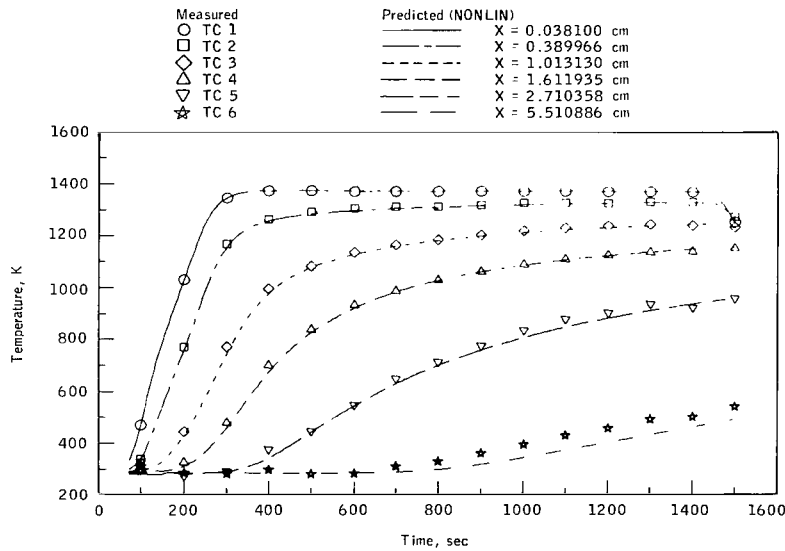


(d) 101 kN/m^2 (760 torr).

Figure 5.- Concluded.

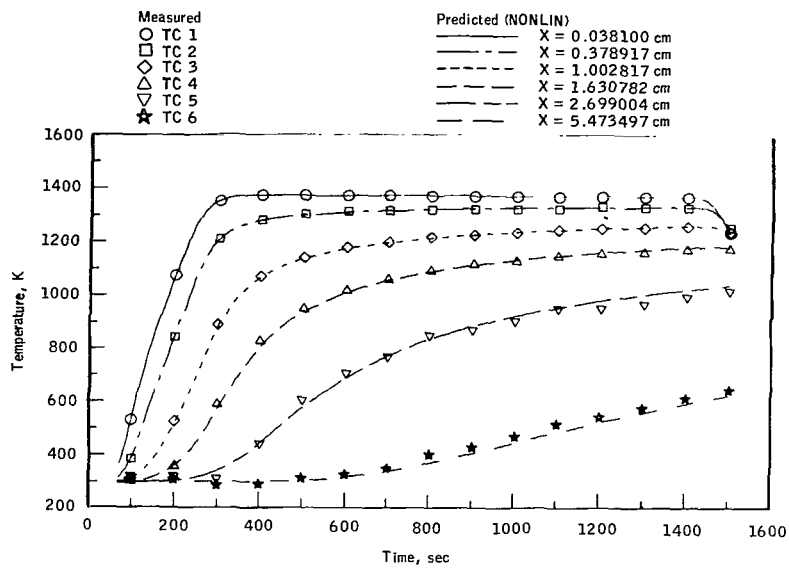


(a) 0.10 kN/m² (0.76 torr).

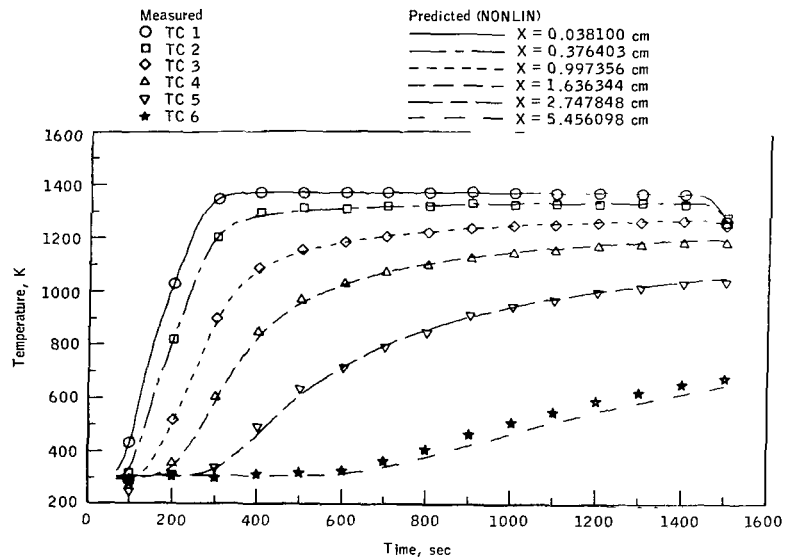


(b) 1.0 kN/m² (7.6 torr).

Figure 6.- Comparison of temperature response using predicted thermal conductivity from specimen RB37 tested at various pressures for relocated-thermocouple locations at depth X (curves) with measured thermocouple values (data points).

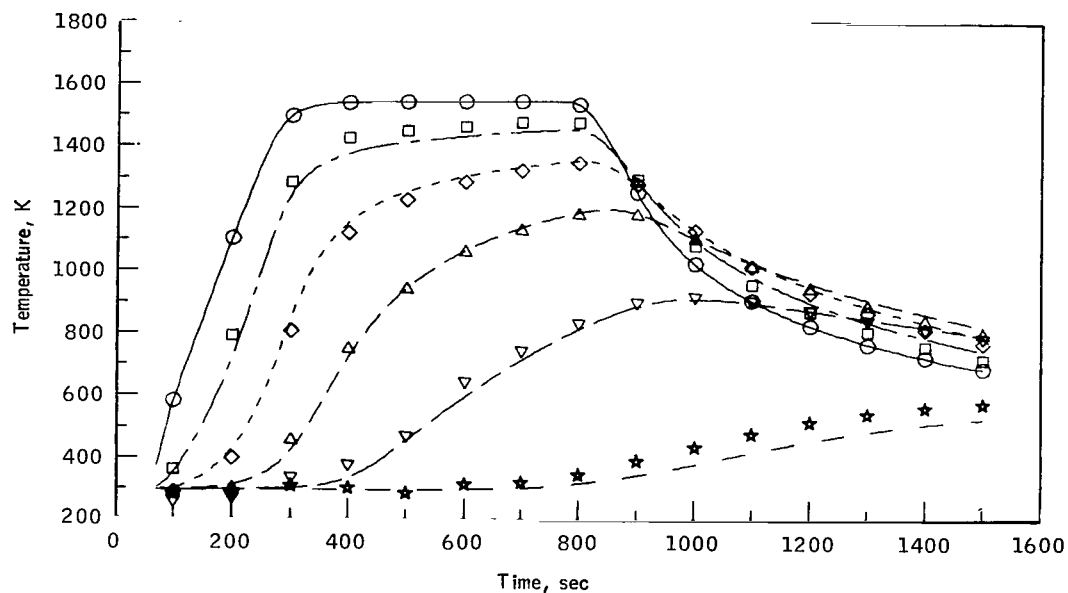


(c) 10 kN/m² (76 torr).

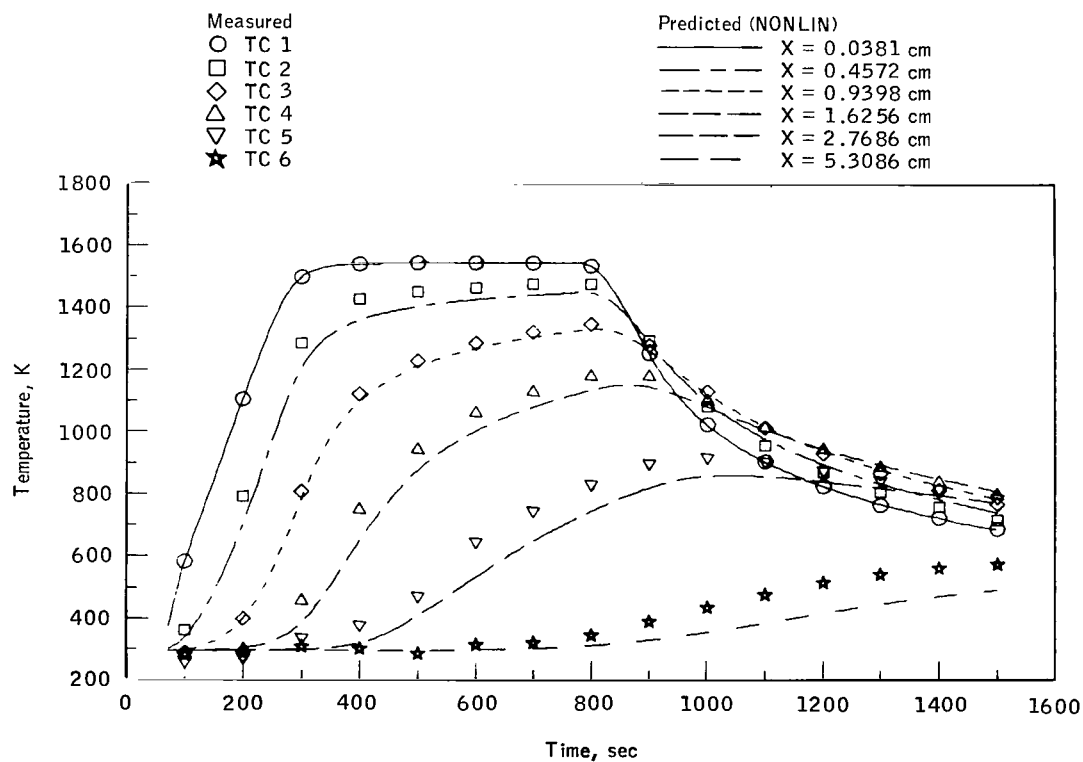


(d) 101 kN/m² (760 torr).

Figure 6.- Concluded.

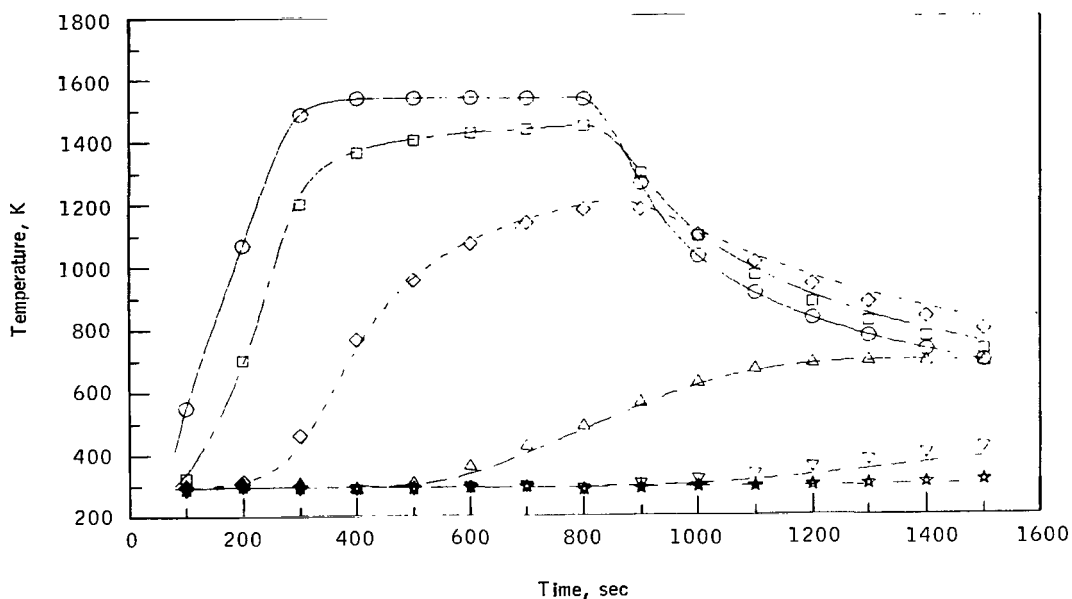


(a) Relocated-thermocouple thermal conductivity.

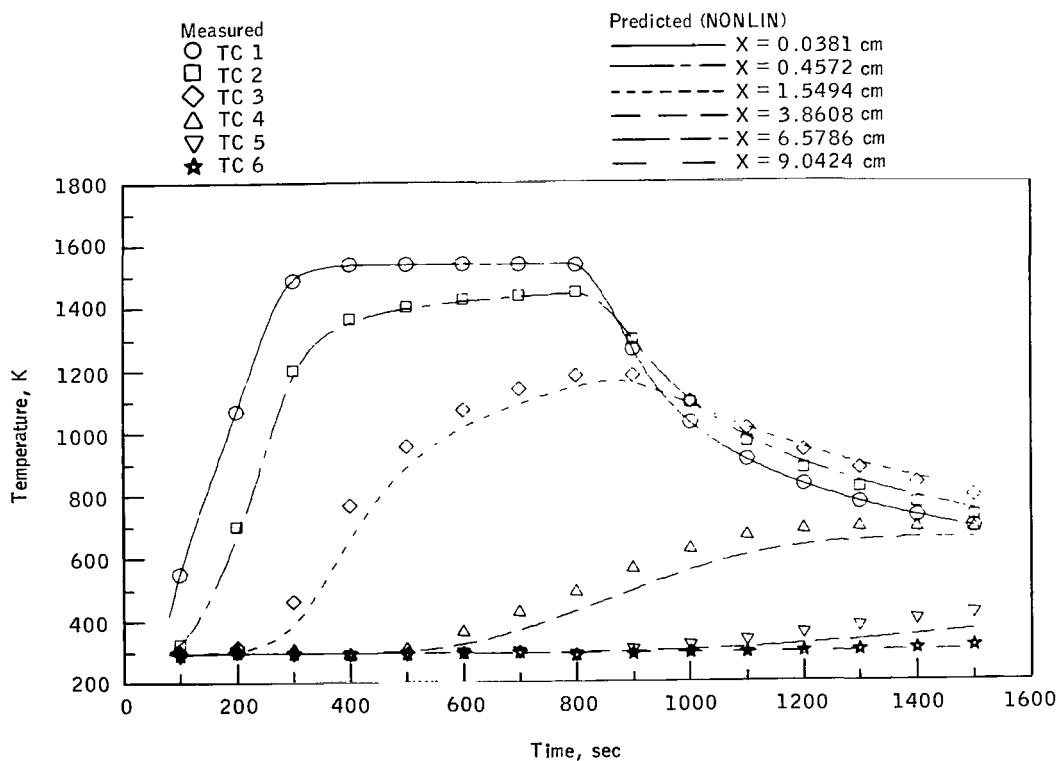


(b) Baseline thermal conductivity.

Figure 7.- Comparison of mission profile temperature response at nominal thermocouple locations (curves) with measured values (data points) for specimen RB37.



(a) Relocated-thermocouple thermal conductivity.



(b) Baseline thermal conductivity.

Figure 8.- Comparison of mission profile temperature response at nominal thermocouple locations (curves) with measured values (data points) for specimen B5892.

2. In-depth radiation effects are not fully accounted for under GHP test conditions but are closely approximated in the analytical model using transient data.

3. The thermal conductivity values determined either through GHP or by analytical calculations are not exact, but the uncertainty in GHP values is probably greater than or equal to the uncertainties associated with the analytical calculations.

4. The analytical calculations represent the best predictions over the entire temperature range at all times as opposed to isolated discrete measurements.

CONCLUDING REMARKS

That nonlinear least squares can be used with confidence to determine effective thermal conductivity from dynamic thermal performance data has been demonstrated. In addition, a method was presented for assessing the relative error associated with the predicted conductivity values.

The thermal conductivity for a low-density, high-porosity insulation material was predicted from four constant-temperature/constant-pressure tests. It was demonstrated that the standard error of temperature and the percent error of thermal conductivity could be reduced by 50 percent through analytical relocation of the thermocouple positions in the thermal model. Whereas the predicted data were 18 percent higher than guarded-hot-plate data, the error analysis indicated that there was less than a 6-percent relative error for a 3σ confidence level in the analytically predicted data.

An analysis of the thermal response for a simulated Space Shuttle mission profile was made using both predicted and guarded-hot-plate thermal conductivity values for two separate test specimens. These tests demonstrated that more reliable results can be obtained using analytically determined thermal conductivities.

This report also points out the differences between dynamic (transient) and static (guarded-hot-plate) determination of effective thermal conductivity where the means of heat transfer in the insulation material is by a combination of conduction, convection, and radiation.

Lyndon B. Johnson Space Center
National Aeronautics and Space Administration
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16. Abstract That nonlinear least squares can be used to determine effective thermal conductivity is demonstrated, and a method for assessing the relative error associated with these pre-dicted values is provided. The differences between dynamic and static (guarded hot plate) determination of effective thermal conductivity of low-density materials that transfer heat by a combination of conduction, convection, and radiation are discussed.				13. Type of Report and Period Covered Technical Paper	
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